Docket No.: 259431US0PCT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF: GROUP: 1651
Hiroto KIKUCHI, et al.

SERIAL NO: 10/516,307 EXAMINER: KOSAR, A. J.

FILED: December 10, 2004

FOR: PROCESS FOR FURIFICATION OF DIFRUCTOSE DIANHYDRIDE III

DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

Sir:

Now comes Hiroto Kikuchi who deposes and states:

1. That I am a graduate of Hokkaido University and received a master's degree in the year 1993.

2. That I have been employed by Nippon Beet Sugar Mfg., Co, Ltd., for 16 years as a researcher in the field of food chemical.

3. That I understand the English language or, at least, that the contents of the Declaration were made clear to me prior to executing the same.

4. That the following experiments were carried out by me or under my direct supervision and control.

5. DFA-III produced by the method of the invention were compared to DFA-III produced by otherwise similar methods using much larger activated carbon particles (Sample B) or no carbon particles at all (Sample C). Comparative sensory testing was carried out on each sample.

6. Sample Preparation. Samples A, B and C were compared. Sample A (active carbon average particle size about 35 microns) corresponds to the invention and Sample B (larger

carbon particles about 1,000 microns) and Sample C (no active carbon) are controls.

Sample A (present invention): Inulin is dissolved in 1 kg of hot water at 80°C, and cooled down to 60 °C. To the resulting solution is added IFT 5000 units/kg inulin, and the mixture is stirred at 60°C for 24 hours to yield a DFA III solution.

The reaction mixture is heated up to 80°C to deactivate the enzyme.

To this deactivated solution is added Taiko Active Charcoal S (Futamura Kagaku Kogyo KK; **average particle size about 35 microns**) at a rate of **1%** to the solid content, and the mixture is stirred at 60°C for 10 minutes. The mixture was then filtered through diatomaceous earth (Showa Chemical Ind.; Radiolite 700) to remove said active carbon.

The filtrate is concentrated in an evaporator at a concentration of R-Bx (Refractometric Brix) 77. To the resulting condensate, the seed crystals are added at 60°C with agitating. The liquid containing the seed crystals is cooled to 20°C for 20 hours with agitating, and then, further kept to cool at 20°C for 3 hours. The resulting crystallized mother liquor is divided into crude crystals and crude crystal syrup by a separator. The crude crystals are redissolved. The resulting redissolved solution is subjected to active carbon treatment, filtration treatment through diatomaceous earth, concentration, crystallization and separation, in a similar manner as described above, to yield a crystal product.

Sample B (treatment with active carbon average particle size 1,000 microns): The DFA III containing solution is prepared in a similar manner as the Sample A described above.

The reaction mixture is heated up to 80°C to deactivate the enzyme.

To this deactivated solution is added granular active carbon (product of Firm Y; average particle size about **1,000 microns**) at a rate of 1% to the solid content, and the mixture is stirred at 60°C for 10 minutes. The mixture was then filtered through diatomaceous earth (Showa Chemical Ind.; Radiolite 700) to remove said active carbon.

The filtrate is concentrated in an evaporator at a concentration of R-Bx (Refractometric Brix)77. To the resulting condensate, the seed crystals are added at 60°C with agitating. The liquid containing the seed crystals is cooled to 20°C for 20 hours with agitating, and then, further kept to cool at 20°C for 3 hours. The resulting crystallized mother liquor is divided into crude crystals and crude crystal syrup by a separator. The crude crystals are redissolved. The resulting redissolved solution is subjected to active carbon treatment, filtration treatment through diatomaceous earth, concentration, crystallization and separation, in a similar manner as described above, to yield a crystal product.

Sample C (treatment without addition of active carbon): The DFA III containing solution is prepared in a similar manner as the Sample A described above.

The reaction mixture is heated up to 80°C to deactivate the enzyme, but **no activated carbon is added**.

The resulting deactivated solution is then filtered through diatomaceous earth (Showa Chemical Ind.; Radiolite 700).

The filtrate is concentrated in an evaporator at a concentration of R-Bx (Refractometric Brix) 77. To the resulting condensate, the seed crystals are added at 60°C with agitating. The liquid containing the seed crystals is cooled to 20°C for 20 hours with agitating, and then, further kept to cool at 20°C for 3 hours. The resulting crystallized mother liquor is divided into crude crystals and crude crystal syrup by a separator. The crude crystals are redissolved. The resulting redissolved solution is subjected to filtration treatment through diatomaceous earth, concentration, crystallization and separation, in a similar manner as described above, to yield a crystal product.

7. Sensory Testing was conducted on DFA-III crystal samples A, B and C. A panel of 20 expert panelists visually and olfactorily scored the color and odor of each sample. Samples determined to have little or no color or odor were given a high score of "3", those having some color or odor a score of "2" and samples with high relative color or odor a low score of "1". The Table below shows the average score for each of Samples A, B and C.

	Color average score of expert panel	Smell average score of expert panel
Sample A (invention; average size of active carbon particles ~35 μm)	2.95	2.80
Sample B (average size of active carbon particles = 1,000 μm)	2.05	2.20
Sample C (no active carbon)	1.00	1.00

8. As apparent from the results above, Sample A produced by the method of the invention had a **295%** better color and **280%** better odor in comparison to Sample C which had no active carbon added. Moreover, in comparison to Sample B, which used active carbon

having an average particle size of 1,000 μm, Sample A had a **144%** better color and **127%** better odor.

9. In addition to the testing results above, 19 out of 20 expert panelists rated Sample A as having no color and 16 out of 20 rated it as having no odor.

10. As apparent from the results shown above, the method used to produce Sample A (according to the invention) produces a crystalline DFA III product having superior color and a lack of odor in comparison to the methods used to make Samples B and C.

11. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

12. Further deponent saith not.

Hiroto Kikuchi
Signature

January 15, 2010
Date

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(OSMMN 05/06)